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6. AUTHOR(S) Sy-Hwang Liou and Roger D. Kirby				
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13. ABSTRACT (Maximum 200 words) We studied and synthesized magnetic single domain particles, improved the resolution of magnetic force microscopy (MFM) tips for magnetic domain imaging and studied both experimentally and theoretically the magnetization reversal process in nanostructured materials. CoPt thin films with well-separated 100-200nm crystallites of well-ordered fct structure were achieved by controlling the annealing time and annealing temperature. Our CoPt films show magnetic coercivity up to 37kOe which is the highest coercivity ever observed in this system. We used CoPt for coating magnetic force microscopy tips. We observed better-resolved magnetic images of a reference recording disk taken with CoPt MFM tips. We also developed advanced magnetic force microscopy tips using focused ion beam milling to obtain higher resolution in magnetic images. We have shown that the magnetization reversal behavior depends strongly on the magnetic field dependence of the energy barrier to thermally-activated reversal. The model calculations carried out thus far predict temperature-dependent "activation volumes" similar to those measured in such materials as CoSm thin particulate films and Co/Au multilayers. These studies have significant implications for high temperature magnetic stability and for long-term stability of magnetic data storage media.				
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## Statement of the Problem Studied

This project has been focused on:

- (1) The study and fabrication of magnetic single domain particles.
- (2) The improvement of the resolution of magnetic force microscopy (MFM) tips for magnetic domain imaging.
- (3) Experimental and theoretical studies of the magnetization reversal process in nanostructured materials.
- (4) Magnetic domain images by magnetic force microscopy with an applied magnetic field.

## Summary of the most important results

### (1) Enhancement of Coercivity in Nanometer-Size CoPt Crystallites

Many magnetic devices are required to have high magnetic coercivity ( $H_c$ ), such as magnetic bias films of magneto-resistive elements, magnetic tips for magnetic force microscopy, high-density magnetic recording media and magneto-optic recording media. The CoPt binary alloy is an excellent system because of its chemical stability and high magnetic anisotropy. The anisotropy of CoPt compounds is as high as  $4 \times 10^7$  ergs/cm<sup>3</sup> and the saturation magnetization is about 800 emu/cm<sup>3</sup>.

For this study, we showed that CoPt thin films with well-separated crystallites and well-ordered fct structure were achieved by controlling the annealing time and annealing temperature. From atomic force microscopy and magnetic force microscopy studies, the magnetic single domain size of CoPt is in the range of 100 to 200 nm. The high  $H_c$  is likely due to the well-separated nanometer-size crystallites and the well-ordered fct phase of CoPt alloy. For the first time, we have enhanced the magnetic coercivity of a CoPt film to 37 kOe.

### (2) CoPt Magnetic Force Microscopy (MFM) Tips

The magnetization direction of a magnetic force microscopy (MFM) tip will change during measurement if the magnetic coercivity ( $H_c$ ) of the MFM tip is lower than the magnetic stray field of the sample. As a result, the magnetic images are difficult to interpret. This is also a problem in the determination of the magnetization direction of the MFM tip, when the demagnetization field can not be neglected due to the shape of the tip. These problems can be resolved by using high  $H_c$  MFM tips because its  $H_c$  is higher than the demagnetization field.

To our knowledge, we have fabricated CoPt MFM tips with high  $H_c$  for the first time. We demonstrated that the  $H_c$  of these MFM tips is higher than the gap field of a typical thin-film recording head. The magnetic transitions and magnetic grains in a recording disk were also clearly observed. This was likely due to the better defined magnetization direction and very small size of the high  $H_c$  CoPt MFM tip. There are possibly many other advantages in the application of the high  $H_c$  tips. For example, the high  $H_c$  tips may be used in the study of magnetic domain images in the presence of an external magnetic field. It has also been shown by Proksch et al. that the power dissipation in the magnetic image can be eliminated by using a high  $H_c$  CoPt MFM tip which will result in improved images.

### (3) Advanced Magnetic Force Microscopy Tips

We have developed several methods to fabricate advanced MFM tips for magnetic images. In this work, we used our combined resources in thin film preparation, tip microfabrication techniques and expertise in magnetic force microscopy.

Magnetic force microscopy (MFM) is a useful imaging tool for studying a wide variety of local magnetic phenomena. It allows the direct visualization of magnetic domains and provides the experimental basis for theoretical modeling. The technique measures changes in the interaction force between a magnetized probe and the local stray magnetic field from the sample,

point by point, as the probe is scanned across the surface. The probe is typically a cantilever made from silicon or silicon nitride, with a ferromagnetic tip on the free end. The inherent resolution depends upon the confinement of the interaction at the end of the probe and sensitivity depends upon the ratio of the cantilever spring constant to the magnetic moment. At present, commercial MFM probes resolve about 50-100 nm features at force constant of about 0.01 N/m -- roughly equivalent to resolving the field gradients from a  $10^{-12}$  emu source at a distance of 50 nm. Despite the impressive performance and widespread use of the MFM, there are important probe-related limitations that need to be overcome to realize its full potential. The first of these is the enhancement of resolution and sensitivity. As is well known from microscopy, in order to measure something at a given length scale it is necessary to have a probe whose dimensions are much smaller than those of the object to be measured. In the case of magnetic force microscopy (MFM) the force on the magnetic probe is determined by the stray field gradient arising from the sample under investigation. Obviously, the smaller the magnetically active volume of the probe, the less it will be affected by areas from far away since the dipole nature of the field causes it to diminish rapidly with distance. Therefore, in order to make a high resolution MFM it is necessary to create an extremely small magnetic probe. Fig. 1 shows a few examples of the advanced magnetic force microscopy tips that were made in our laboratory by different methods.

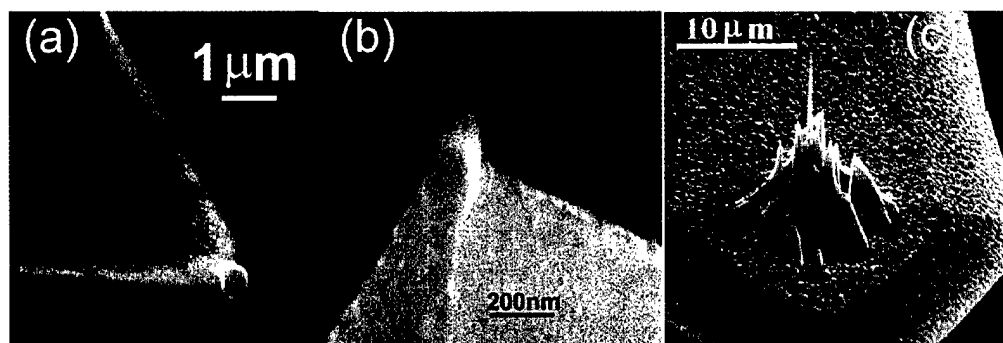


Fig. 1 Advanced magnetic force microscopy tips made by three different methods (a) by ion milling, (b) electron beam deposition, (c) focused ion beam milling.

We have successfully fabricated a very sharp MFM tip. The tip was first coated with a magnetic film then machined by a focused ion beam source (in collaboration with Professor Jon Orloff at the University of Maryland), so that there is a nanometer-size magnetic particle on the very end of the tip. The smaller size of the magnetic particle result in much improved lateral resolution. Fig. 2 shows the domain configuration of a 150nm-thick epitaxial (110) Fe film obtained by a MFM tip that was machined by a focused ion beam source.

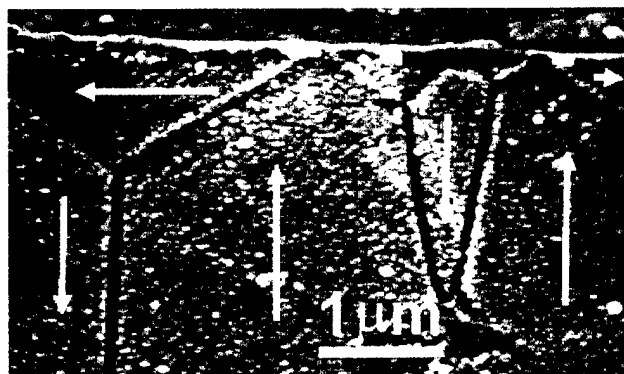


Fig. 2 The domain configuration of a 150nm-thick epitaxial (110) Fe film obtained by a MFM tip that was machined by a focused ion beam source.

The image was obtained with a vertically magnetized tip in a zero applied magnetic field to the sample. As shown in the above magnetic images the MFM tip innovations undoubtedly improved imaging capability. The arrows indicate the magnetization direction. The full width at half maximum of the Bloch domain wall width was measured to be 60-70nm, which agrees well with the calculated value for that of bulk Fe. The above MFM tip innovations undoubtedly improved imaging capability. We will continue a systematic investigation of this advanced tip technology.

#### **(4) Magnetic Domain Images by Magnetic Force Microscopy with an Applied Magnetic Field**

The dependence of the domain structure of thin magnetic films on the size of the sample, its thickness, shape, and the effect of the underlying layer on the domain structure can now be imaged in the presence of an applied magnetic field using magnetic force microscopy. We will be able to improve the performance of magnetic devices by better understanding the response of magnetic domains under magnetic fields.

The magnetization direction of a magnetic force microscopy (MFM) tip will change during measurement if the magnetic coercivity ( $H_c$ ) of the MFM tip is lower than the stray magnetic field of the sample. As a result, the magnetic images are often difficult to interpret. This is also a problem in the determination of the magnetization direction of the MFM tip, when the demagnetization field can not be neglected due to the shape of the tip. These problems can be resolved by using high  $H_c$  MFM tips because its  $H_c$  is higher than the demagnetization field.

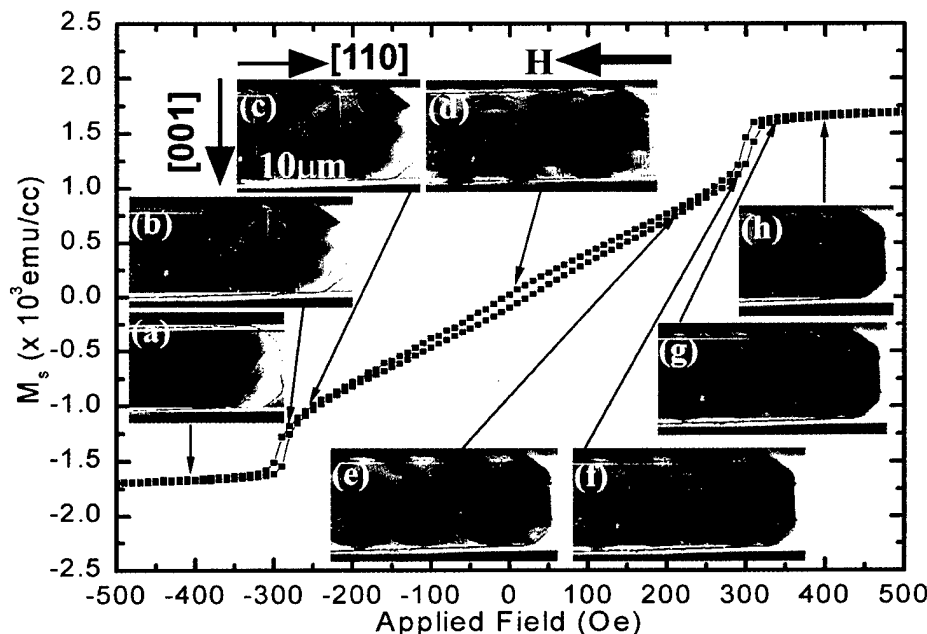


Fig.3 Magnetic domain structures in a patterned epitaxial (110) Fe film under a magnetic field. The film was magnetized at  $-5,000$  Oe before the measurement. (a) at  $-400$  Oe, the edge domain wall is formed, (b) between  $-300$  Oe and  $-270$  Oe the domain wall propagates from the edge into the sample. It shows a rapid change of magnetization (c) at  $-260$  Oe the domain structure is formed in the film. (c) (d) (e) (f) between  $-260$  Oe and  $260$  Oe, the domain walls only move a short distance. This shows flux closure domains. (g) at  $300$  Oe the domain wall starts to disappear and domain walls exist only at the edge and at the surface (the surface domain wall has a much lower signal i.e. light color.) (h) at  $400$  Oe, the sample is nearly saturated, however, the surface domain wall is still visible. In contrast, there is no surface domain in the domain pattern (a) (that was magnetized at  $-5,000$  Oe before the measurement). The surface domain disappears at an applied magnetic field of about  $600$  Oe.

To our knowledge, we have fabricated CoPt MFM tips with high  $H_c$  for the first time. We demonstrated that the  $H_c$  of these MFM tips is higher than the gap field of a typical thin-film recording head. The magnetic transitions and magnetic grains in a recording disk were also

clearly observed. This was due to the better defined magnetization direction and very small size of the high  $H_c$  CoPt MFM tip. There are many other advantages in the application of the high  $H_c$  tips. For example, the high  $H_c$  tips may be used in the study of magnetic domain images in the presence of an external magnetic field. An example of this type study is shown in Fig. 3. In this case, the magnetic field was applied along the hard axis [110] of a patterned epitaxial (110) Fe film. The sample has a  $10\text{ }\mu\text{m}$  width,  $200\text{ }\mu\text{m}$  length, and  $100\text{ nm}$  thickness. The magnetization curve was measured by a SQUID magnetometer. The detailed domain evolution can clearly be seen, showing that we are able to directly correlate the magnetic domain patterns with the magnetization curve.

### **(5) Magnetization Reversal Process**

We have studied both experimentally and theoretically the magnetization reversal process in nanostructured materials with the goal of better understanding reversal mechanisms at high temperatures. For most magnetic systems at high temperature, reversal occurs when the local magnetization of a particle or grain gains enough random thermal energy to “hop” over the anisotropy energy barrier, or for a domain wall to jump over a pinning energy barrier. Such thermally-activated processes dominate at room temperature and above and must be realistically modeled. We have developed a simple model of interacting particles (both exchange and dipolar interactions) which permits us to simulate the reversal process over a wide range of temperatures and applied magnetic fields.

One characteristic measure of thermally-activated reversal is the so-called “activation volume”. The activation volume  $V^*$  is determined by measuring the sweep rate dependence of the coercivity or by carrying out magnetic relaxation measurements of various kinds.  $V^*$  is often used to estimate whether the magnetization of a magnetic material is stable to thermal fluctuations. Researchers often assume that magnetization reversal occurs by coherent rotation of the moments (which may be approximately true for particulate systems with very small magnetic particles). A key feature of the coherent rotation reversal process is that the energy barrier to thermal activation varies approximately as  $E_B = E_0 - M_s V$ , where  $M_s$  is the saturation magnetization and  $V$  is the particle volume. In this case, simulations lead to the conclusion that the activation volume is approximately equal to the particle volume. However, it is quite unlikely that reversal in nanoscale materials occurs by coherent rotation of the magnetization. A variety of calculations describing nucleation at particle surfaces or domain wall pinning indicate that  $E_B$  varies roughly as  $1/H$ , where  $H$  is the magnetic field – a very different field dependence of the energy barrier. We have applied these ideas to a number of magnetic systems, including magnetic recording media like CoCrPt and magnetic multilayers like Co/Pt.

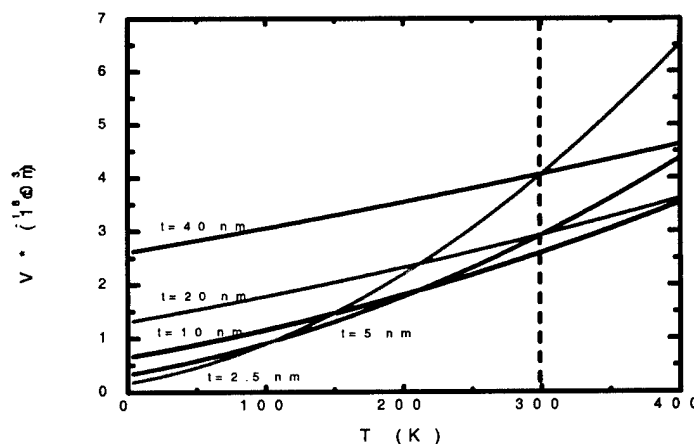


Fig. 4:  $V^*$  as a function of temperature for several different particle heights ( $t$ ). Note in particular that  $V^*$  varies as  $T^2$  at high temperature.

Our result indicates that the *measured* activation volume (as modeled by simple systems) depends critically on the field dependence of  $E_B$ . If  $E_B$  varies linearly with  $H$ , then one expects the activation volume and the magnetic grain volume to be similar. If however,  $E_B$  varies as  $1/H$ , then the high temperature activation volume is in fact inversely proportional to the magnetic grain volume. Figure 4 shows the simulated activation volumes as a function of temperature for several different particle volumes. Note in particular that at low temperatures, the activation volumes are much smaller than the particle volume, but much larger at high temperatures. Further,  $V^*$  increases like  $T^2$  at high temperatures. These results are qualitatively in agreement with measurements on CoCrPt particulate films.

## **(6) Fabrication and Characterization of Nanostructured Magnetic Clusters**

These nanostructured thin films can be formed using a number of techniques, including electron beam patterning, stamping and replication, holographic lithography, and others. As examples, magnetic domain patterns of  $\text{Fe}_{20}\text{Ni}_{80}$  magnetic thin films with different lateral dimensions are shown in Fig. 5. The rectangular patterns of the thin films were made by e-beam lithography. This clearly shows that the domain patterns depend on both the size and shape of the film. It shows that the elongated shape film do not have a single magnetic domain. This observation is direct evidence that other factors (such as stress, crystalline defects, etc) may play important role in the nanometer-size magnetic films. The schematic diagrams shown at the left of Fig.5 indicate that the cross-tie-domain.

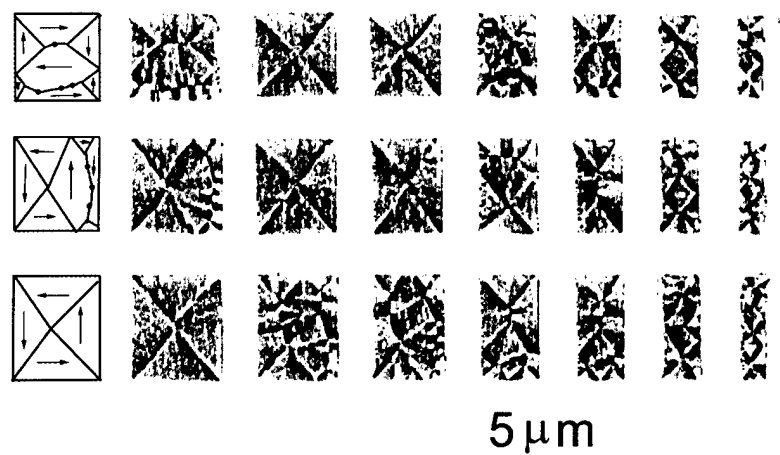


Fig.5 Array of micrometer to submicro-meter Permalloy islands showing domain configurations as a function of the aspect ratio. The rectangular patterns of the thin films were made by e-beam lithography.

## **Publications and Presentations**

### **(a) Papers published in peer-reviewed journals:**

1. Y.Liu, Richard A. Thomas, S. S. Malhotra, Z.S. Shan, S.H. Liou and D.J. Sellmyer; "Phase Formation and Magnetic Properties of in Co-Rare Earth Magnetic Films", J. Appl. Phys., 83, 6244(1998).
2. M. Yu, Y. Liu, S. H. Liou and D. J. Sellmyer; "Nanostructured NdFeB Films Processed by Rapid Thermal Annealing" J. Appl. Phys., 83, 6611(1998).
3. S. H. Liou, and Y. D. Yao; "Development of High Coercivity Magnetic Force Microscopy Tips" Journal of Magnetism and Magnetic Materials, 190, 130(1998).

4. S. H. Liou, S. Huang, E. Klimek, R. D. Kirby, Y. D. Yao; "Enhancement of Coercivity in Nanometer-Size CoPt Crystallites" J. Appl. Phys. 85, 4334(1999)
5. S. H. Liou, "Comparison of Magnetic Images Using Point and Thin-Film Magnetic Force Microscopy Tips", IEEE Trans. on Magnetics, 35, 3989 (1999).
6. Roger D. Kirby, Mingjun Yu, and D. J. Sellmyer, *Activation Volumes in Thin Film and Particulate Systems*, J. Appl. Phys., 87, 5696 (2000)
7. C. P. Luo, S. H. Liou, and D. J. Sellmyer, "FePt:SiO<sub>2</sub> Granular Thin Films for High Density Magnetic Recording", J. Appl. Phys. 87, 6941 (2000).
8. C. P. Luo, S. H. Liou, L. Gao, Y. Liu and D. J. Sellmyer; "Nanostructured FePt:B<sub>2</sub>O<sub>3</sub> Thin Films with Perpendicular Magnetic Anisotropy", Appl. Phys. Lett, 77, 2225 (2000).

**(b) Papers published in conference proceedings:**

1. A. Gavrin, C. Sellers, S. H. Liou; "Magnetic Domain Observation on Melt-Spun Nd-Fe-B Ribbons Using Magnetic Force Microscopy" Mat. Res. Soc. Symp. Proc. Vol. 577, 241(1999).

**(c) Papers submitted for publications:**

1. M. L. Yan, S. H. Liou, and D. J. Sellmyer, Y. Liu, and M. J. Yu; "Magnetic properties of Nanostructures Co<sub>x</sub>Pt<sub>100-x</sub>:C Thin Films", submitted to J. Appl. Phys. (2000)
2. M. Zheng, M. Yu, R. Skomski, S. H. Liou, D. J. Sellmyer, N. I. Polushkin; "Magnetic Nanodot Arrays Produced by Direct Laser Interference Lithography", submitted to Appl. Phys. Lett. (2000)
3. S. H. Liou, R. F. Sabiryanov, S. S. Jaswal, J. C. Wu and Y. D. Yao; "Magnetic Domain Patterns of Rectangle and Ellipse Arrays of Small Permalloy Elements" accepted by J. of Magnetism and Magnetic Materials, (2000).

**(d) Thesis:**

1. S. Huang, "The Magnetic Properties of CoPt Thin Films" M. S. Thesis at the University of Nebraska, 1998.

**(e) Invited Talks**

1. S. H. Liou, "Advanced Magnetic Force Microscopy Tips for Magnetic Characterization", Presented at International Symposium on Advanced Magnetic Technologies, Taipei, Taiwan, May 24-25, 1999.
2. S. H. Liou, "Magnetic Domains of Nanometer-Size Magnetic Features", Presented at University of Missouri-Kansas City, October 8, 1999.
3. S. H. Liou, "Magnetic Domain Images of Nanometer Size Magnetic Features" October 6, 2000 University of Indiana, Bloomington Indiana.

**(f) Presentations:**

1. Y. Liu, Richard A. Thomas, S. S. Malhotra, Z.S. Shan, S. H. Liou, and D.J. Sellmyer; "Phase Formation and Magnetic Properties of Co-Rare Earth Magnetic Films", Presented at The 7<sup>th</sup> Joint MMM-Intermag Conference, San Francisco, California, January 6-9, 1998.
2. M. Yu, Y. Liu, S.H. Liou, and D.J. Sellmyer; "Nanostructured Nd-Fe-B Films Processed by Rapid Thermal Annealing", Presented at The 7<sup>th</sup> Joint MMM-Intermag Conference, San Francisco, California, January 6-9, 1998.

3. S. H. Liou, and Y. D. Yao; "Development of High Coercivity Magnetic Force Microscopy Tips" Presented at MFM-10: Using the Magnetic Force Microscope in Research and Industry, San Francisco, California, January 9-11, 1998.
4. Sa Huang and S. H. Liou; "High Magnetic Field Coercivity of Single-Domain Particles" Bull. of American Physical Society, Vol. 43, 716(1998).
5. S. Huang, S. H. Liou, E. Klimek, R. D. Kirby, Y. D. Yao; "Enhancement of Coercivity in Nanometer-Size CoPt Crystallites" Presented at 43rd Annual Conference on Magnetism and Magnetic Materials, Miami, Florida, November 9-12, 1998.
6. Roger D. Kirby; "The Interpretation of Activation Volumes in Magnetic Viscosity Measurements" Presented at March 1999 meeting of American Physical Society, Atlanta GA, 1999.
7. Roger D. Kirby, Mingjun Yu, and D. J. Sellmyer, *Activation Volumes in Thin Film and Particulate Systems*, Presented at the 44<sup>th</sup> Annual Conference on Magnetism and Magnetic Materials, San Jose, CA, November 14-18, 1999.
8. A. Gavrin, C. Sellers, S. H. Liou; "Magnetic Domain Observation on Melt-Spun Nd-Fe-B Ribbons Using Magnetic Force Microscopy" presented at Material Research Society Spring meeting, San Francisco, California, April 5-9, 1999.
9. Victor Souw, Michael McElfresh, and Sy-Hwang Liou; "Magnetic Mapping of MFM Tips Using Miniature Hall Sensor Arrays", Bull. of American Physical Society, Vol. 44, 542, (1999).
10. S. H. Liou, "Comparison of Magnetic Images Using Point and Thin-Film Magnetic Force Microscopy Tips", Presented at The 1999 IEEE International Magnetic Conference, Kyogju, Korea, May 18-21, 1999.
11. Chunping Luo, Sy-Hwang Liou, and David J. Sellmyer, "FePt:SiO<sub>2</sub> Granular Thin Films for High Density Magnetic Recording", Presented at 44th Annual Conference on Magnetism and Magnetic Materials, San Jose, California, November 15-18, 1999.
12. Victor Souw, Mike McElfresh, and Sy-Hwang Liou; "Magnetic Mapping of MFM Tips Using Miniature Hall Sensor Arrays", Bull. of American Physical Society, Vol. 44, 542(1999).
13. S. H. Liou, "Comparison of Magnetic Images Using Point and Thin-Film Magnetic Force Microscopy Tips", Presented at International Magnetic Conference, Kyogju, Korea, May 18-21, 1999. .
14. Chunping Luo, Y. Liu, S. H. Liou, and D. J. Sellmyer, "FePt:B<sub>2</sub>O<sub>3</sub> Nanocomposite Thin Films with perpendicular Magnetic Anisotropy", Bull. of American Physical Society, Vol. 45, 618(2000)
15. S. H. Liou, R. F. Sabiryanov, S. S. Jaswal, J. C. Wu and Y. D. Yao; "Magnetic Domain Patterns of Rectangle and Ellipse Arrays of Small Permalloy Elements", Presented at International Conference on Magnetism, August 6-11, 2000.
16. M. L. Yan, S. H. Liou, and D. J. Sellmyer, Y. Liu, and M. J. Yu; "Magnetic properties of Nanostructures Co<sub>x</sub>Pt<sub>100-x</sub>:C Thin Films", Presented at 8<sup>th</sup> Joint MMM-Intermag Conference, San Antonio, Texas, January 7-11, 2001.

## List of all Participants

### Students:

1. Steven Michalski ( graduate student).
2. Sa Huang (graduate student).
3. Elizabeth Klimmek (was an undergraduate student, now is an graduate student).
4. Lan Gao (graduate student).
5. LeighAnn Nicholl (graduate student).
6. Chad Petersen (undergraduate student).
7. Jason Sneed (undergraduate student).



**Senior Personnel:**

1. Sy-Hwang Liou (PI)
2. Roger D. Kirby (Co-PI)
3. Ming-Te Liu (Postdoctoral)

**Student Placement**

1. S. Huang received a M. S. degree in August.
2. Elizabeth Klimek received a B. S. degree in August, 1999..

**Cooperative Laboratory Interactions**

1. High coercivity magnetic force microscopy tips were sent to Professor Joseph Garbini's group at the University of Washington for a magnetic resonance force microscopy study.
2. We are collaborating with Drs. Ken Bobcock and Peter Nelson at Digital Instruments and Professor Jon Orloff at the University of Maryland to develop advanced magnetic force microscopy tips for magnetic characterization.
3. High coercivity magnetic force microscopy tips were sent to Professor Robert P. Ferier's group at the University of Glasgow, United Kingdom for a stray magnetic field mapping study.
4. High coercivity magnetic force microscopy tips were sent to Dr. Kannan M. Krishnan's group at Ernest Orlando Lawrence Berkeley National Laboratory for magnetic force microscopy studies
5. High coercivity magnetic force microscopy tips were sent to Dr. John Moreland at National Institute of Standard and Technology for a magnetic resonance force microscopy study.
6. High coercivity magnetic force microscopy tips were sent to Professor Gomez Romel's group at the University of Maryland for magnetic force microscopy studies
7. High coercivity magnetic force microscopy tips were sent to Dr. Liesl Folks's group at IBM for magnetic force microscopy studies. We also signed a joint study agreement (agreement No. 2325)
8. High coercive magnetic force microscopy tips were sent to Professor Andrew Kent's group at the New York University for magnetic force microscopy studies
9. High coercive magnetic force microscopy tips were sent to Professor Conradin Beeli's group at the Swiss Federal Institute of Technology Zurich (ETH Zurich) Switzerland for electron holography studies.
10. High coercive magnetic force microscopy tips were sent to Professor Jin Shi's group at the Utah University for magnetic force microscopy studies